

PATENT COOPERATION TREATY

PCT

INTERNATIONAL PRELIMINARY REPORT ON PATENTABILITY

(Chapter II of the Patent Cooperation Treaty)

(PCT Article 36 and Rule 70)

Applicant's or agent's file reference 72085/ÅD	FOR FURTHER ACTION See Form PCT/IPEA/416	
International application No. PCT/SE2003/001076	International filing date (<i>day/month/year</i>) 23.06.2003	Priority date (<i>day/month/year</i>)
International Patent Classification (IPC) or national classification and IPC B01D 61/48		
Applicant VATTENFALL AB et al		

<p>1. This report is the international preliminary examination report, established by this International Preliminary Examining Authority under Article 35 and transmitted to the applicant according to Article 36.</p> <p>2. This REPORT consists of a total of <u>5</u> sheets, including this cover sheet.</p> <p>3. This report is also accompanied by ANNEXES, comprising:</p> <p style="margin-left: 20px;">a. <input checked="" type="checkbox"/> (<i>sent to the applicant and to the International Bureau</i>) a total of <u>3</u> sheets, as follows:</p> <div style="margin-left: 40px;"> <input checked="" type="checkbox"/> sheets of the description, claims and/or drawings which have been amended and are the basis of this report and/or sheets containing rectifications authorized by this Authority (see Rule 70.16 and Section 607 of the Administrative Instructions). </div> <div style="margin-left: 40px;"> <input type="checkbox"/> sheets which supersede earlier sheets, but which this Authority considers contain an amendment that goes beyond the disclosure in the international application as filed, as indicated in item 4 of Box No. I and the Supplemental Box. </div> <p style="margin-left: 20px;">b. <input type="checkbox"/> (<i>sent to the International Bureau only</i>) a total of (indicate type and number of electronic carrier(s)) _____, containing a sequence listing and/or tables related thereto, in electronic form only, as indicated in the Supplemental Box Relating to Sequence Listing (see Section 802 of the Administrative Instructions).</p>																									
<p>4. This report contains indications relating to the following items:</p> <table style="width: 100%; border: none;"> <tr> <td style="width: 10%;"><input checked="" type="checkbox"/></td> <td style="width: 20%;">Box No. I</td> <td>Basis of the report</td> </tr> <tr> <td><input type="checkbox"/></td> <td>Box No. II</td> <td>Priority</td> </tr> <tr> <td><input type="checkbox"/></td> <td>Box No. III</td> <td>Non-establishment of opinion with regard to novelty, inventive step and industrial applicability</td> </tr> <tr> <td><input type="checkbox"/></td> <td>Box No. IV</td> <td>Lack of unity of invention</td> </tr> <tr> <td><input checked="" type="checkbox"/></td> <td>Box No. V</td> <td>Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement</td> </tr> <tr> <td><input type="checkbox"/></td> <td>Box No. VI</td> <td>Certain documents cited</td> </tr> <tr> <td><input type="checkbox"/></td> <td>Box No. VII</td> <td>Certain defects in the international application</td> </tr> <tr> <td><input checked="" type="checkbox"/></td> <td>Box No. VIII</td> <td>Certain observations on the international application</td> </tr> </table>		<input checked="" type="checkbox"/>	Box No. I	Basis of the report	<input type="checkbox"/>	Box No. II	Priority	<input type="checkbox"/>	Box No. III	Non-establishment of opinion with regard to novelty, inventive step and industrial applicability	<input type="checkbox"/>	Box No. IV	Lack of unity of invention	<input checked="" type="checkbox"/>	Box No. V	Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement	<input type="checkbox"/>	Box No. VI	Certain documents cited	<input type="checkbox"/>	Box No. VII	Certain defects in the international application	<input checked="" type="checkbox"/>	Box No. VIII	Certain observations on the international application
<input checked="" type="checkbox"/>	Box No. I	Basis of the report																							
<input type="checkbox"/>	Box No. II	Priority																							
<input type="checkbox"/>	Box No. III	Non-establishment of opinion with regard to novelty, inventive step and industrial applicability																							
<input type="checkbox"/>	Box No. IV	Lack of unity of invention																							
<input checked="" type="checkbox"/>	Box No. V	Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement																							
<input type="checkbox"/>	Box No. VI	Certain documents cited																							
<input type="checkbox"/>	Box No. VII	Certain defects in the international application																							
<input checked="" type="checkbox"/>	Box No. VIII	Certain observations on the international application																							

Date of submission of the demand 14.01.2005	Date of completion of this report 29.09.2005
Name and mailing address of the IPEA/SE Patent- och registreringsverket Box 5055 S-102 42 STOCKHOLM Facsimile No. +46 8 667 72 88	Authorized officer Jens Waltin/MP Telephone No. +46 8 782 25 00

INTERNATIONAL PRELIMINARY REPORT ON PATENTABILITY

International application No.

PCT/SE2003/001076

Box No. I Basis of the report

1. With regard to the **language**, this report is based on:

- ☒ the international application in the language in which it was filed
- ☐ a translation of the international application into _____, which is the language of a translation furnished for the purposes of:
- ☐ international search (Rules 12.3(a) and 23.1(b))
- ☐ publication of the international application (Rule 12.4(a))
- ☐ international preliminary examination (Rules 55.2(a) and/or 55.3(a))

2. With regard to the **elements** of the international application, this report is based on (*replacement sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this report as "originally filed" and are not annexed to this report*):

- ☐ the international application as originally filed/furnished
- ☒ the description:
- pages 1 - 17 as originally filed/furnished
- pages* _____ received by this Authority on _____
- pages* _____ received by this Authority on _____
- ☒ the claims:
- pages _____ as originally filed/furnished
- pages* _____ as amended (together with any statement) under Article 19
- pages* 1 - 3 received by this Authority on 26.09.2005
- pages* _____ received by this Authority on _____
- ☒ the drawings:
- pages 1 - 3 as originally filed/furnished
- pages* _____ received by this Authority on _____
- pages* _____ received by this Authority on _____
- ☐ a sequence listing and/or any related table(s) – see Supplemental Box Relating to Sequence Listing.

3. ☐ The amendments have resulted in the cancellation of:

- ☐ the description, pages _____
- ☐ the claims, Nos. _____
- ☐ the drawings, sheets/figs _____
- ☐ the sequence listing (*specify*): _____
- ☐ any table(s) related to the sequence listing (*specify*): _____

4. ☐ This report has been established as if (some of) the amendments annexed to this report and listed below had not been made, since they have been considered to go beyond the disclosure as filed, as indicated in the Supplemental Box (Rule 70.2(c)).

- ☐ the description, pages _____
- ☐ the claims, Nos. _____
- ☐ the drawings, sheets/figs _____
- ☐ the sequence listing (*specify*): _____
- ☐ any table(s) related to the sequence listing (*specify*): _____

* If item 4 applies, some or all of those sheets may be marked "superseaded."

INTERNATIONAL PRELIMINARY REPORT ON PATENTABILITY

International application No.

PCT/SE2003/001076

Box No. V Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

1. Statement

Novelty (N)	Claims	<u>1-16</u>	YES
	Claims		NO
Inventive step (IS)	Claims	<u>1-16</u>	YES
	Claims		NO
Industrial applicability (IA)	Claims	<u>1-16</u>	YES
	Claims		NO

2. Citations and explanations (Rule 70.7)

This report is based upon the amended claims filed with the letter of 26-09-2005.

Documents cited in the International Search Report:

D1: US 4148708 A
D2: EP 1075868 A2
D3: WO 02096807 A2
D4: WO 03031034 A1

D1 represents the most relevant prior art.

D1 relates to a combination of ion exchange and electrodialysis apparatuses to process water from the primary coolant loop of a nuclear power plant. The process produces purified water as well as concentrated boric acid and lithium hydroxide.

The differences between the invention and D1 are:

1. The process according to the invention is carried out in at least four chambers versus three chambers in D1.
2. According to the invention, cations and strongly dissociated anions are separated from the feed solution in the same chamber, and boron species are separated in another chamber. In D1, on the other hand, boron species and cations are separated in the same chamber, while any competitive anions are separated from the boron solution in another chamber.
3. The invention provides for recirculation of anolyte and catholyte, while there is no recirculation in D1.

.../...

Supplemental Box

In case the space in any of the preceding boxes is not sufficient.

Continuation of: BOX V

It is not considered obvious to a person skilled in the art to modify the process known from D1 so as to arrive at the invention defined in claim 1.

As for the other cited documents, D2 and D3 both relate to an electro-deionisation apparatuses used for removing weakly ionised species such as boron, silica from water, in order to produce ultra-pure water.

D4 relates to a water treatment system including a boron analyser and a control system for controlling the performance of a boron removal unit. The boron removal unit may be an electro-deionisation treatment unit.

The water treatment systems of D2-D4 are all aimed at producing purified water, and not at the recovery of boron. Therefore, none of D2-D4 provides purification of the separated boron solution from other anions, which is an important feature of the present invention. This can be seen for example in D3, where a common reject line is provided for all separated ions. Furthermore, the invention differs from D2-D4 by other constructional features of the electrochemical cell, such as the number of chambers and recirculation of anolytes/catholytes.

It is not considered obvious to a person skilled in the art to modify the technique disclosed in any of D2-D4 so as to arrive at the invention defined in claim 1.

The conclusion is that the claimed invention is novel and presents an inventive step over D1-D4. It is also considered to be industrially applicable.

Box No. VIII Certain observations on the international application

The following observations on the clarity of the claims, description, and drawings or on the question whether the claims are fully supported by the description, are made:

Claim 1 is inconsistent with an embodiment disclosed in the description, and does thus not fulfil the requirements of PCT Article 6.

It follows from claim 1, that the process is carried out in at least four compartments (two diluted compartments, a catholyte compartment and at least one anolyte compartment). Furthermore, one diluted compartment is filled with cation-exchange material ONLY.

On the other hand, according to the simplified embodiment shown in fig. 2 and page 9, lines 6-10, the process is carried out in only three compartments. Moreover, there is only one diluted compartment, which should be filled with anion- AND cation-exchange material.

Re: PCT/SE/03/01076

IAP9 Rec'd PCT/PTO 07 DEC 2005

Amended claims

1. A process for the separation and recovery of boron from aqueous solutions containing the same which aqueous solutions are used in nuclear power generation, comprising separating strongly dissociated anions in the form of electrical migration performed in one diluted compartment (2) of an electrochemical cell, filled with cation-exchange material only; separating dissociated cations such as ${}^7\text{Li}^+$ in the form of ion-exchange/electrical migration in the same compartment above; separating boron in the form of electrochemical/chemical dissociation, ion-exchange/adsorption, and electrical migration performed in another diluted compartment (5) filled with anion-exchange material or a mixture of anion- and cation-exchange materials or layers of anion- and cation-exchange materials separated from each other; recovering the separated cations into a catholyte compartment (1) of the electrochemical cell; recovering the separated boron into at least one anolyte compartment (3,4) of the electrochemical cell; recirculating the anolyte in the at least one anolyte compartment (3,4); recirculating the catholyte in the catholyte compartment (1); and recirculating the diluted solution in the diluted compartments (2,5) if necessary.
2. The process according to claim 1, wherein a five-compartment electrochemical cell is used comprising two diluted compartments (2,5), two anolyte compartments (3,4), one catholyte compartment (1), one anode and two cathodes.
3. The process according to claim 1, wherein the diluted compartments (2,5) are separated from the anode by an anion-exchange membrane (AM) and separated from one of the cathodes by a cation-exchange membrane (CM).
4. The process according to any of the preceding claims, wherein DC potential is applied between the anode and the cathode.
5. The process according to any of claims 1-4, wherein one anolyte compartment (3) is used for collecting the separated strongly dissociated anions, such as chloride, nitrate and sulfate, and another anolyte compartment (4) is used for recovering the separated boron.

6. The process according to any of the preceding claims, wherein the catholyte compartment (1) is used for collecting the separated cations such as ${}^7\text{Li}^+$.
7. The process according to any of the preceding claims, wherein the initial anolyte is the pure solution of boric acid, and the initial catholyte is the pure solution of a given cation that may be recovered, and the initial concentrations of the anolyte and catholyte are appropriately adjusted for performing the separation and recovery of boron and a certain cation.
8. The process according to any of the preceding claims, wherein the ion-exchange materials filled in the diluted compartment(s) is (are) ion-exchange resins having uniform particle size and the same mean diameter of resin beads for both anion and cation resins.
9. The process according to any of the preceding claims, wherein the separation of boron from strongly dissociated anions is performed before the separation of boron in a following diluted compartment.
10. The process according to any of the preceding claims, wherein the electrochemical dissociation of boric acid in the first diluted compartment is reduced by controlling the density of DC current during the separation of boron with strongly dissociated anions, the applied current density being controlled below 0.1 A/dm^2 , and the electrochemical dissociation of boric acid is reduced below 15% as the initial concentration of boron is about 2000 ppm.
11. The process according to any of the preceding claims, wherein the DC current applied to the electrochemical cell is appropriately adjusted to keep a balance among the electrochemical dissociation of boric acid, the electrical migration of anions and water splitting for the regeneration of ion-exchange materials.
12. The process according to any of the preceding claims, wherein the separation and recovery of boron can be performed for an aqueous solution with a wide range of initial concentration of boron from several thousands ppm to several tens ppm.
13. The process according to any of the preceding claims, wherein a high efficiency of boron separation is achieved, the separation percentage of boron being over 95%.

14. The process according to any of the preceding claims, wherein a high concentrating limit is achieved for boron recovery, the concentration of boron in the anolyte being up to 80% of the solubility of boric acid.

15. The process according to any of the preceding claims, wherein the separation and recovery of boron and a given cation like lithium such as ${}^7\text{Li}^+$ may be performed at the same time.

16. The process according to any of the preceding claims, wherein the treatment of the aqueous solution is performed in a recirculating model, a follow-through model or a partial-recirculating model.